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# Adiabatic Compression Sensitivity of Liquid Fuels and Monopropellants

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## KEYWORDS

Adiabatic compressibility, explosions, hazard sensitivity, propellants, fuels, oxidizers

## ABSTRACT

Liquid rocket fuels and monopropellants can be sensitive to rapid compression. Such liquids may undergo decomposition and their handling may be accompanied with risk. Decomposition produces small gas bubbles in the liquid, which upon rapid compression may cause catastrophic explosions. The rapid compression can result from mechanical shocks applied on the tank containing the liquid or from rapid closure of the valves installed on the lines. It is desirable to determine the conditions that may promote explosive reactions.

At AFRL, we constructed an apparatus and established a safe procedure for estimating the sensitivity of propellant materials towards mechanical shocks (Adiabatic Compression Tester). A sample is placed in a stainless steel U-tube, held isothermally at a temperature between 20 and 150 °C then exposed to an abrupt mechanical shock of nitrogen gas at a pressure between 6.9 and 20.7 MPa (1000 to 3000 psi). The apparatus is computer interfaced and is driven with LABTECH NOTEBOOK-pro ® Software. In this presentation, the design of the apparatus is shown, the operating procedure is outlined and the safety issues are addressed. The results obtained on different energetic materials will be presented.

## INTRODUCTION

Considerable efforts have been directed towards the study of the sensitivity and stability of liquid fuels and rocket propellants. One of the safety issues concerned is the rapid compression of these liquids if, for example, their containers are exposed to mechanical shocks during transportation. Another concern arises when a liquid fuel or propellant is transferred from the storage tank through pipelines to the combustion chamber of the rocket. If the lines contain entrained air bubbles, fuel vapor bubbles, or even

bubbles generated from propellant decomposition, the rapid closure or opening of online valves may cause a sudden compression of the liquid and, as a result, an explosion may occur. Furthermore, pumping fuels at considerably high flow rates, as it is required in many rocketry applications, can cause cavitation and may introduce vapor bubbles into the liquid.

In general, when vapor bubbles are in contact with the liquid, they act as hot spots; the mechanical work of rapid compression is converted into thermal energy given to the system. The thermal energy causes the molecules in the bubbles to thermally decompose with the generation of additional amounts of heat (most of the decomposition reactions are typically exothermic in nature). The generated heat will further decompose the liquid present at the vicinity of the bubbles and a chain of chemical decomposition reactions begins. These processes take place in a few milliseconds and may eventually end up with an explosion. In the case of air bubbles trapped within the liquid fuel, a similar process will occur but the reaction can initiate via a different route. The first step is the combustion (or oxidation) of the fuel with concurrent liberation of heat. The generated heat will promote the decomposition of additional amounts of the fuel with subsequent liberation of heat. When the chain reactions continue, an explosion may occur.

We have constructed an apparatus to evaluate the adiabatic compressibility of liquids. The apparatus has been used to test rocket fuels and propellants. Briefly, the liquid is placed in a stainless steel U-tube which is then held isothermally at a chosen temperature. The propellant is compressed (slammed) with a charge of nitrogen gas at high pressure: 6.90 – 20.684 MPa (1000-3000 psi). If the U-tube ruptures, the result is positive (+) but if it does not, the result is negative (-). For a given propellant, the main variables that affect the results are: type of material from which the U-tube is made, propellant temperature, starting pressure of compressing gas, compression ratio inside the U-tube, and compression rate (psi/s). This rate depends on the speed by which the valve, connecting the U-tube to the high-pressure source, is opened.

In this presentation, we illustrate the schematic diagram and outline the components of the apparatus, constructed at AFRL. The operating procedure, including the safety issues, is summarized. The effects of different variables on test results (+ or –) are discussed. The threshold temperature and pressure limits for which an explosion occurs are given for selected propellants. A comparison is made to evaluate the relative sensitivity of different fuels and propellants when compressed under similar chosen test conditions. As previously stated, liquid propulsion system operation (e.g., valve actuation) can produce high-pressure transients within fuel lines. Depending on the particular scenario, these transients can range from insignificantly small to the order of 7 MPa. Thus, adiabatic compressibility test conditions are chosen with driving pressures at a magnitude reflecting a relatively severe transient. Also, it is necessary to perform adiabatic compressibility tests on liquid propellant at relatively high temperatures. High temperature can be encountered in operations and system designs that allow heat transfer from the combustion chamber and injector to the propellant lines.

## APPARATUS

The schematic diagram of the adiabatic compression apparatus is shown in Figure 1. The system is placed in an isolated test cell. Nitrogen gas (purity better than 99.99%) is supplied from K-bottles at 41.368 MPa (6000 psi) to the system via a hand regulator, HR-1, connected to a hand valve, HV-1, and a

high precision electronic regulator, PR-1. The maximum operating pressure for all tests was chosen at 20.684 MPa (3000 psi); the relief valve, RV-1, was thus set at 22.063 MPa (3200 psi). An adiabatic test was performed at 6.895 MPa, 13.79 MPa or 20.684 MPa (1000, 2000 or 3000 psi). A computer, an A/D and I/O digital interfacing boards, located away from the system, are used to control the entire operation. The electronic regulator injects nitrogen to fill a 5-lit 316-SS accumulator using the remotely operated isolation valve, ROV-1. Two electronic pressure transducers, P-2 and P-3, are placed at the inlet and outlet of the accumulator. A second safety relief valve, RV-2 (set point = 22.063 MPa) was installed on the accumulator to prevent accidental over pressurization. A rapid opening valve, ROV-2, is installed between the accumulator and the U-tube. This valve has four components: a SS-ball valve and a rapid actuator controlled by a pair of air operated solenoid valves. One of the solenoid valves, (ROV-4), activates the actuator into the "OPEN" position while the other (ROV-5) activates to the "CLOSE" position. Two additional components have been installed between ROV-2 and the U-tube: a check valve, CV-1, and a burst disk (rated at 1000, 2000 or 3000 psi). The check valve (rated at 6000 psi) protects the actuator from the excessively high pressure generated inside the U-tube after compressing a propellant. The burst disk has two functions: it prevents the undesirable vapors from escaping back into the system while the sample is held isothermally at the desired temperature and the disk also increases the rate of pressurization. This issue will be addressed shortly.

A temperature bath, placed on a hydraulic (or electric) jack, is used to maintain the temperature of the U-tube. Originally we used a water bath for heating up to 90 °C, however, this practice had its drawbacks. When the U-tube exploded in the bath, the entire amount of water and propellant spilled over the floor of the test cell and the room became contaminated. The steps required to clean and decontaminate the room were time consuming. The water bath was then replaced by a heating bath filled with chrome-steel metal balls (9 mm OD). A stainless steel cover (3 mm thick) was installed above the bath to serve as a protection plate. The plate has a circular opening (diameter = 75 mm) which is centrally aligned with the center of the bath. The opening is covered with a perforated lid (diameter = 120 mm) which is bolted to the protection plate before the beginning of the test. The diameter of holes in the lid is 5 mm.

Unless otherwise stated, the U-tube was made of 316-SS (6.25 mm OD and 0.7 mm wall thickness). To start a test, a new U-tube was washed with acetone and propanol, dried and filled with a 50/50 mixture of HAN (hydroxyl ammonium nitrate) and AFN (AMINE FUNCTIONAL NITRATE). The tube was baked overnight at 75 °C, washed with distilled water and dried. It was then filled under the hood with 3.0 cc of the propellant. One end of the tube was capped off (using Swagelok fitting) while the other end was connected to the system. A new burst disk (1000, 2000 or 3000 psi) was installed, depending on the target pressure chosen for the sample. The fittings of the entire system were tightened. Attention was paid to insure that the hand operated valve, HV-2 was closed before the start of a run. The empty temperature bath was raised till it rested against the protection plate. The preheated chrome steel balls were introduced through opening of the plate and the perforated lid was secured. A thermocouple, TC-3, was inserted through the lid. The door of the box containing the system was closed and the test cell was evacuated and closed.

Inside the control room, the computer is turned on and a custom-made program, called "Batch-Runner" is automatically loaded. When the operator starts the program, the first screen appears and a series of safety questions and checkup instructions are displayed. When completed, the operating software, LABTECH NOTEBOOK-pro ® and the adiabatic program are downloaded. The second screen shown in

Figure 2 appears which represents the actual control unit of the apparatus. After 10 seconds, a manually operated "ignition switch" is turned on in order to supply the electric power to the valves, regulator, transducers and computer boards. The operator simply follows the 10 steps shown on the screen. The vent valve is closed (step 1), the power to the regulator is turned on (step 2), the lines are pressurized with nitrogen to a target pressure of 6.895 MPa (1000 psi), 13.79 MPa (2000 psi) or 20.684 MPa (3000 psi) – step 3. The regulator is then maintained at a constant pressure (step 4). The accumulator is filled with nitrogen to the target pressure (step 5) and the isolation valve is closed (step 6). The sample is compressed at step 7 when the actuator is activated to the "OPEN" position using ROV-4. In step 8, the actuator is activated to the "CLOSE" position using ROV-5. The accumulator is then vented to the atmosphere through ROV-3 (step 9). To terminate the run, the regulator and pipes are vented (step 10).

The operating parameters, real time, temperatures and pressures are updated on the screen and saved to an Excel file every second (or even as fast as 5 milliseconds for valve calibration). The accumulator pressure P2 indicates whether the test result is positive or negative. For example, if the U-tube does not rupture (negative test), the drop in P2 is insignificant because the accumulator volume (5.0 lit) is considerably larger than the U-tube volume (0.028 lit). The drop in pressure is small because the expansion volume is also small. However, when the tube ruptures (positive test), P2 drops considerably and an audible report is heard. In this case, the test cell is opened and allowed to vent for 15 minutes. Typical pressure readings for positive and negative tests are shown in Figure 3 (a) and (b), respectively.

After terminating the operation, the adiabatic program and LABTECH NOTEBOOK-pro ® software are closed and MS-Excel is automatically loaded to open an electronic log file (third screen for Batch Runner). Here, the operator summarizes test conditions and results as well as adding other necessary comments. After closing this file, the last screen appears which reminds the operator with the shut down procedure and other safety considerations.

The preferred testing protocol for liquid energetic materials is one that begins sample testing at the harshest experimental condition (i.e., highest test pressure and sample temperature). It is the nature of most energetic materials to transition from a situation in which positive response is always obtained under generally harsh test conditions to a region of a mixture of positive and negative responses from specimens tested at a less harsh condition. Eventually an experimental condition is found in which the energetic material will not give a positive response. A valuable objective of the adiabatic compressibility test is to ultimately determine the conditions under which the material is not expected to yield a positive response.

## RESULTS AND DISCUSSION

### Compression Rate

One of the critical parameters in adiabatic compression tests is the rate by which the propellant is pressurized. To estimate this rate, it was essential to determine the time required for attaining the maximum pressure (3000 psi) inside the U-tube. A special program was written to save pressure readings at fast rates, using LABTECH NOTEBOOK-pro ® software. The program runs for a maximum of 20 seconds during which it saves 4000 data points. A pressure transducer was installed at one end of an empty U-tube. The other end of the tube was connected to the system in the usual manner. After the

accumulator was filled with 20.684 MPa (3000 psi), ROV-2 valve was opened and the pressure readings inside the tube were saved every 5 milliseconds (Figure 4). In the first test, a 3000 psi-burst disk was installed on line. The time taken to reach 3000 psi inside the U-tube was 25 milliseconds and the rate of compression was 120,000 psi/s (Figure 4). In the second test, no burst disk was installed. The time taken to reach maximum pressure was longer, 48 milliseconds and the corresponding compression rate was lower, 62,500 psi/s. The comparison shows that the compression rate is doubled when a rupture disk is installed. While an increase of compression rate is expected when using a rupture disk, a somewhat greater difference in compression rates was expected since rupture disks are known to open in a fraction of a millisecond.

**The Impact of Adiabatic Explosions**

Figure 5 (a) shows the shape of a new U-tube (height (top to bottom) = 24 cm and radius of curvature = 7.6 cm). The tubes left after negative compression tests looked essentially the same. However, when a positive test was noted, the U-tubes ruptured in different ways. For example, the tube shown in Figure 5 (b) exploded at one end after the compression of nitromethane at 70 °C and 20.684 MPa. In this particular case, the water bath was used to heat the sample. The impact of explosion caused the tube to bend and twist erratically. Figure 3 (c) shows U-tubes ruptured after testing a new energetic propellant developed at AFRL at different temperatures. In one case, the tube split across its length while in the other case, the U-tube was fragmented into many smaller pieces. The U-tubes were not the only part of the apparatus that suffered from the impact. Occasionally, the check valve could not hold the backpressure generated inside the U-tube and the poppet as well as the poppet stop located inside the valve was damaged. Finally, the impact of explosion on the U-tube was strong enough in one case to break the electric jack supporting the temperature bath. The electric jack was replaced with a heavy duty "1-Ton" mechanical jack.

**Results on Simple Compounds**

Table 1 summarizes the adiabatic results obtained on single compounds including hydrazine, n-propyl nitrate, nitromethane, stabilized hydroxyl ammonium nitrate (S-HAN), and different batches of amine functional nitrate (AFN) salt. The runs listed in this table were all executed at 20.684 MPa. From this table, hydrazine appears to be the most stable species to compression, it gave negative results at temperature as high as 120 °C. The stabilized HAN (tests # 9-12) gave positive results at 95 °C and above but was confirmed to be negative at 90 °C (tests # 12, 13, 14). The S-HAN is less stable than hydrazine to adiabatic compression.

The AFN batch 20 was a relatively fresh sample (1-month-old). It was stable to compression at temperatures as high as 110 °C and 20.684 MPA (3000 psi), test # 15-18. This batch was also used to explore the effect of different materials from which the U-tubes are made (tests # 19-21). That is, instead of using the regular 316-SS tubing (high carbon content), we used 316-SS tubing with low carbon content (with both materials, we kept tube diameter and thickness unchanged). With the low carbon tubing, positive results (explosion) occurred at lower temperatures (80-90 °C). We have then adopted 316-SS high carbon tubing as the standard for our tests.

**Results on AFRL Monopropellants**

Table 2 is representative of data obtained with a few monopropellants developed at AFRL, namely RK-315-E, RK-618-A and RK-315-A. In tests # 1 and 2, the effect of compression pressure is noted on the RK-615-I. At 100 °C, a positive test was noted at 20.684 MPa (3000 psi) but a negative test was noted at 13.79 MPa (2000 psi). Samples RK-315-E and RK-618-A (tests # 4-11) gave negative tests at 20.684 MPa and temperatures as high as 90 °C (or even 100 °C). By contrast, sample RK-315-A was the most sensitive material in the evaluation group. At 20.684 and 13.79 MPa, the U-tubes exploded at temperature as low as 13 °C. With this sample, the only negative test was obtained at 13 °C when the compression pressure was low, 6.895 MPa (1000 psi).

## SUMMARY AND CONCLUSIONS

- A sturdy adiabatic compression apparatus has been constructed and interfaced with a PC computer.
- A safe operating procedure has been established in which samples can be tested at temperatures as high as 145 °C and at pressures up to 20.684 MPa (3000 psi).
- The maximum rate of compressing rocket fuels and propellants is 120,000 psi/s.
- Hydrazine is relatively stable to adiabatic compression when compared to other energetic liquids.
- When compressed, experimental AFRL monopropellants range from relatively insensitive (e.g., RK-315-E and RK-618-A) to relatively sensitive (RK-315-A).

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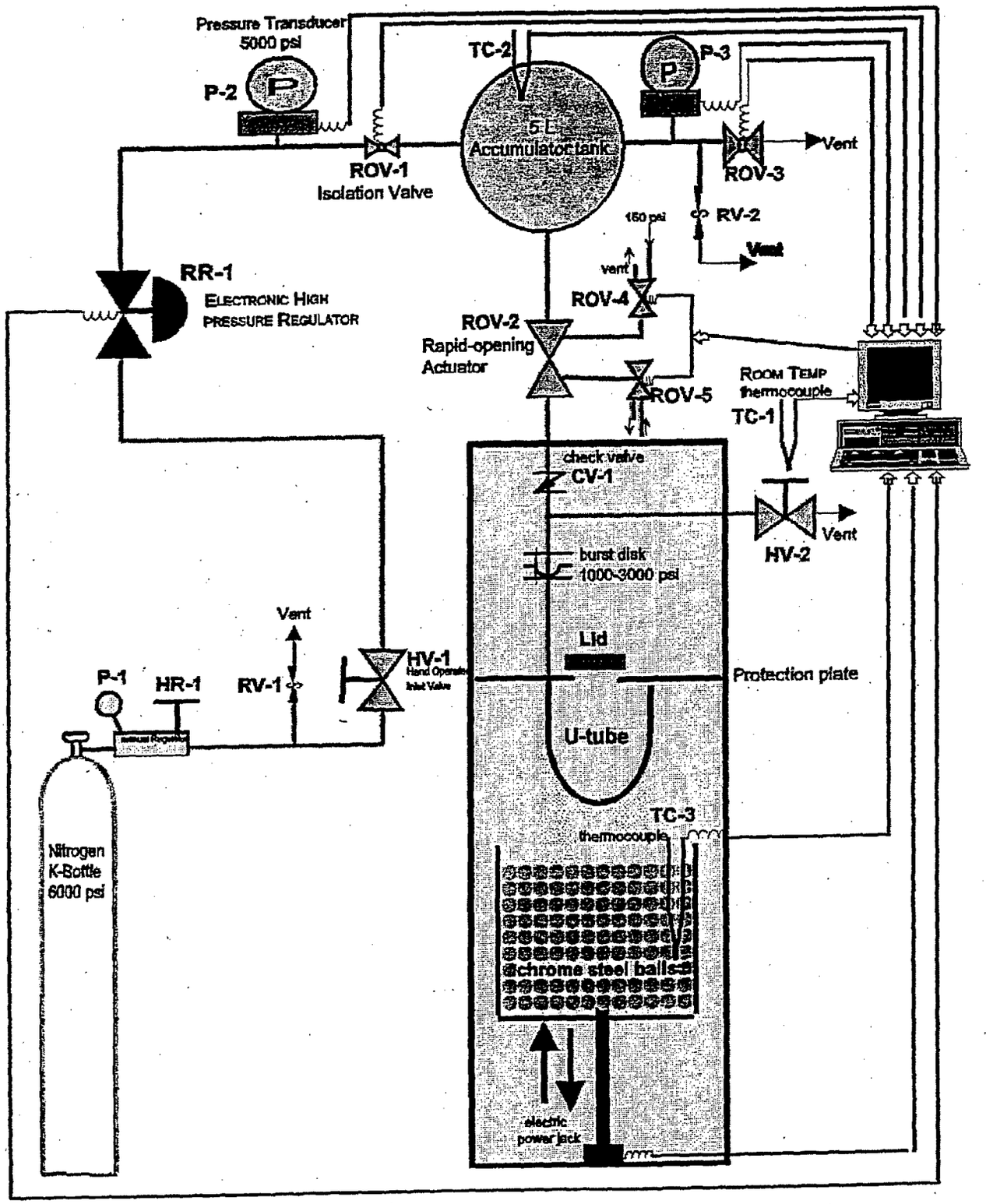



**Figure 1: Apparatus built to study the adiabatic compressibility of liquid propellants.**

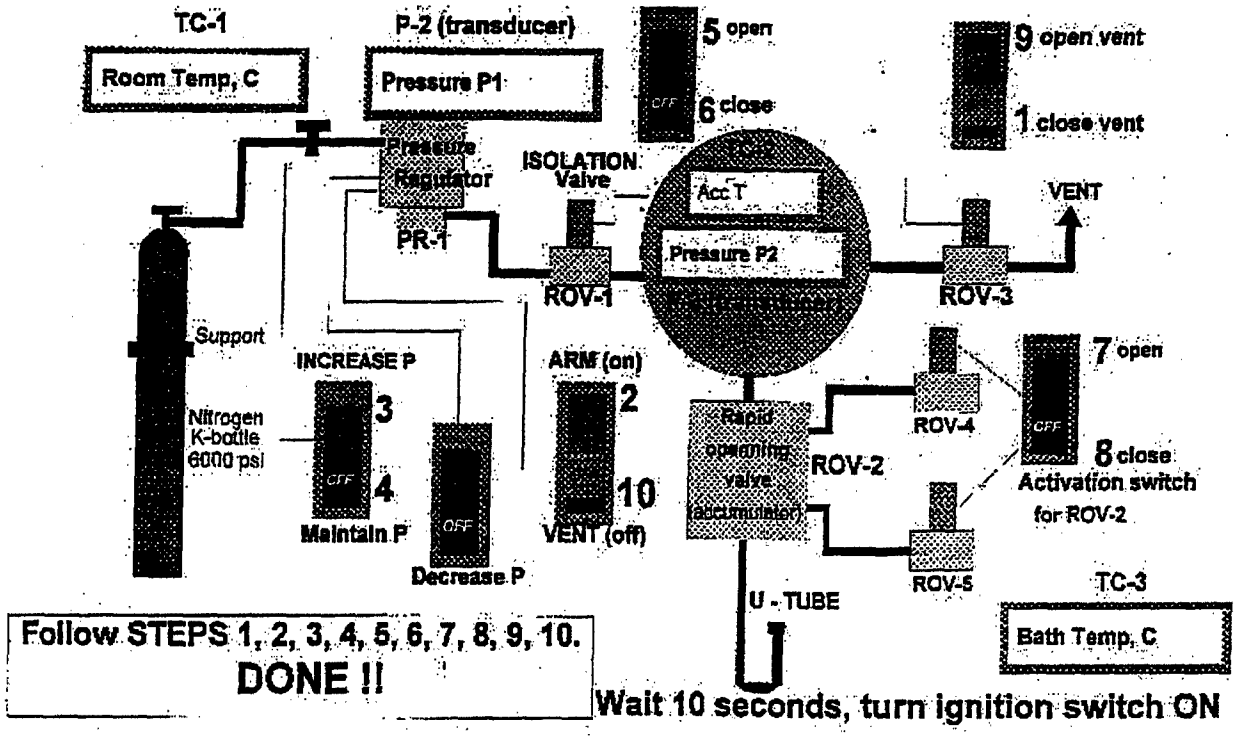



**Figure 2: Control unit operating the adiabatic apparatus.**

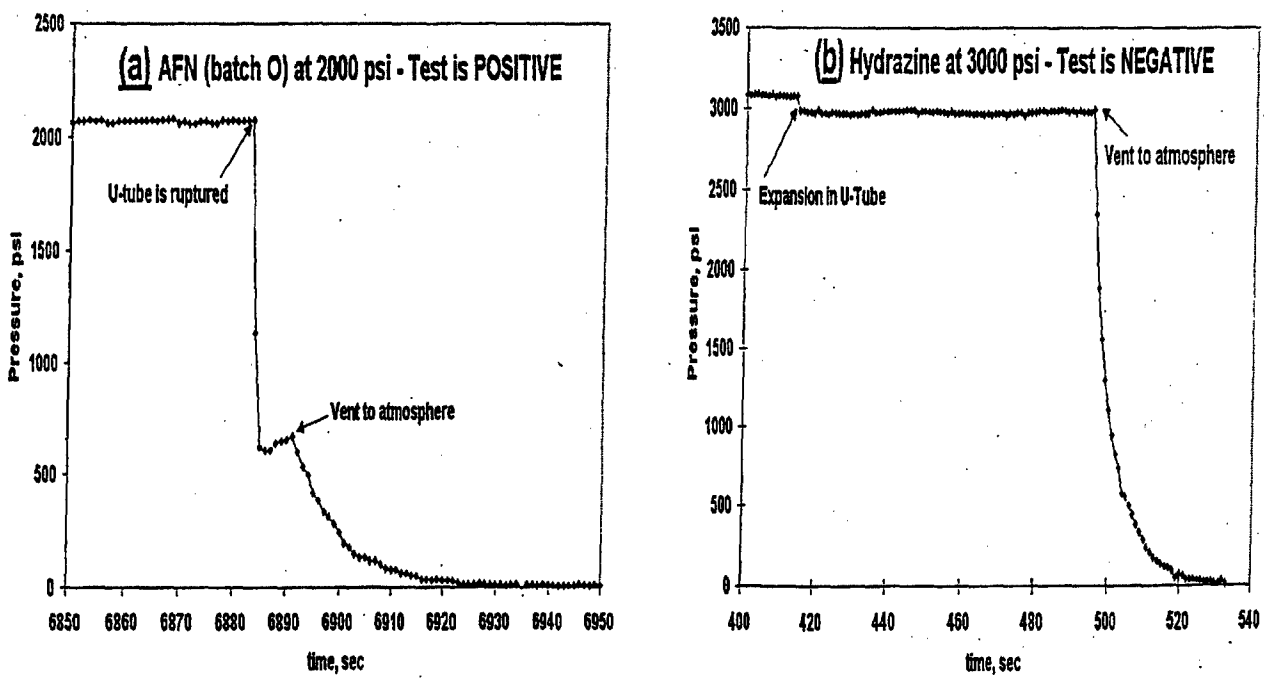



**Figure 3: Typical examples of positive (a) and negative (b) compression tests.**

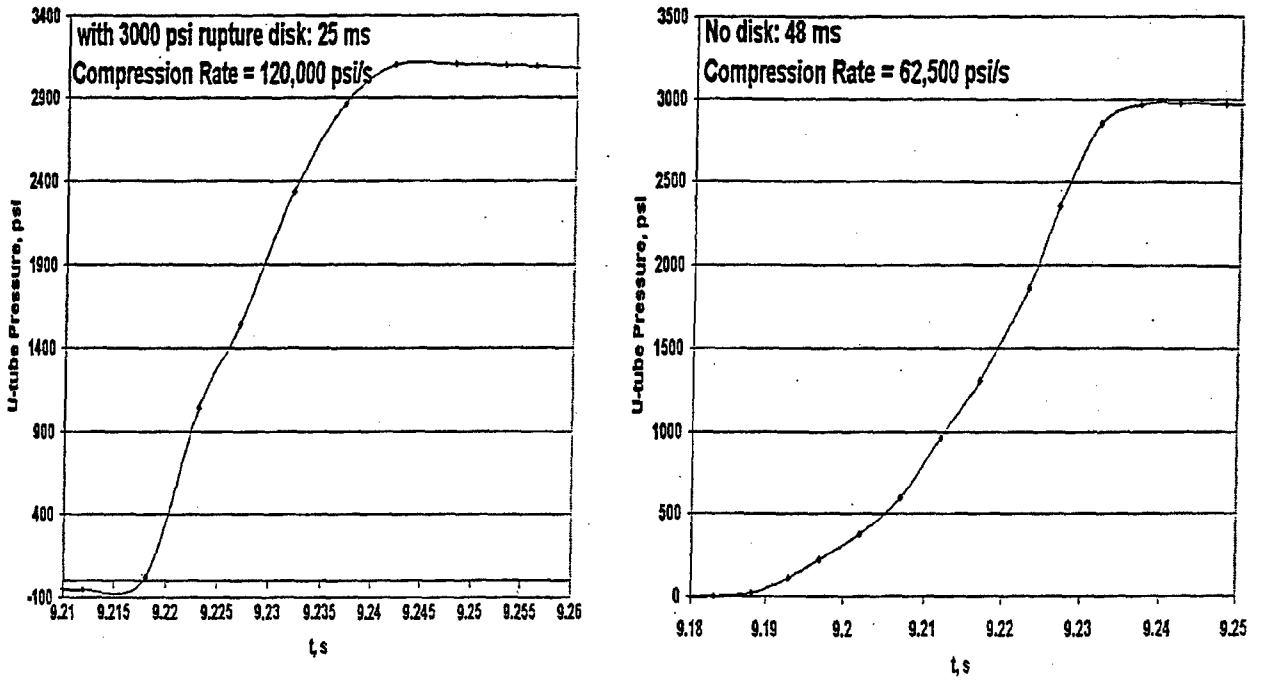
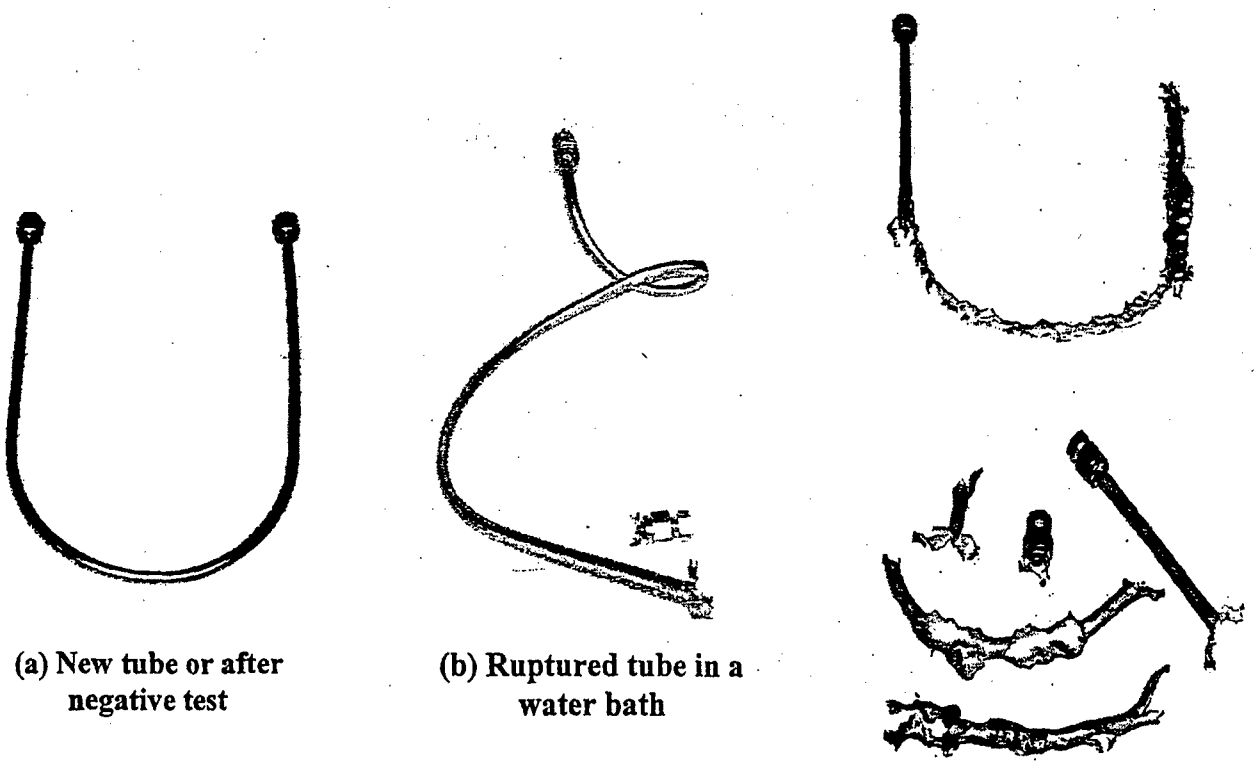



**Figure 4: Compression rates of empty U-tubes with and without a rupture disk.**



(a) New tube or after negative test

(b) Ruptured tube in a water bath

(c) Severely damaged tubes

**Figure 5: New and exploded U-tubes after adiabatic compression.**

**Table 1: Summary of Adiabatic Compression Results Obtained on Different Liquids**

| Test ID # | Sample | Temperature, °C | Pressure, MPa (psi) | Result |
|---|---|---|---|---|
| 1 | Hydrazine | 70 | 20.684 (3000) | - |
| 2 | n-propyl nitrate | 70 | 20.684 (3000) | - |
| 3 | Nitromethane | 70 | 20.684 (3000) | + |
| 4 | Hydrazine | 85 | 20.684 (3000) | - |
| 5 | Hydrazine | 90 | 20.684 (3000) | - |
| 6 | Hydrazine | 100 | 20.684 (3000) | - |
| 7 | Hydrazine | 115 | 20.684 (3000) | - |
| 8 | Hydrazine | 120 | 20.684 (3000) | - |
| 9 | S-HAN-5 | 110 | 20.684 (3000) | + |
| 10 | S-HAN-13 | 110 | 20.684 (3000) | + |
| 11 | S-HAN-13 | 95 | 20.684 (3000) | + |
| 12 | S-HAN-13 | 90 | 20.684 (3000) | - |
| 13 | S-HAN-13 | 90 | 20.684 (3000) | - |
| 14 | S-HAN-13 | 90 | 20.684 (3000) | - |
| 15 | AFN (Batch 20) | 80 | 20.684 (3000) | - |
| 16 | AFN (Batch 20) | 90 | 20.684 (3000) | - |
| 17 | AFN (Batch 20) | 100 | 20.684 (3000) | - |
| 18 | AFN (Batch 20) | 110 | 20.684 (3000) | - |
| 19 | AFN-batch 20 (low carbon 316 SS) | 80 | 20.684 (3000) | + |
| 20 | AFN-batch 20 (low carbon 316 SS) | 85 | 20.684 (3000) | + |
| 21 | AFN-batch 20 (low carbon 316 SS) | 90 | 20.684 (3000) | + |

**Table 2: Summary of Adiabatic Results Obtained on AFRL monopropellants**

| Test ID # | Sample | Temperature, °C | Pressure, MPa (psi) | Result |
|---|---|---|---|---|
| 1 | RK-618-A | 100 | 20.684 (3000) | + |
| 2 | RK-618-A | 100 | **13.79 (2000)** | - |
| 3 | RK-618-A | 90 | 20.684 (3000) | - |
| 4 | RK-618-A | 90 | 20.684 (3000) | - |
| 5 | RK-618-A | 90 | 20.684 (3000) | - |
| 6 | RK-100 | 100 | 20.684 (3000) | - |
| 7 | RK-315 E | 90 | 20.684 (3000) | - |
| 8 | RK-315 E | 90 | 20.684 (3000) | - |
| 9 | RK-315 E | 90 | 20.684 (3000) | - |
| 10 | RK-315 E | 100 | 20.684 (3000) | - |
| 11 | RK-315 E | 100 | 20.684 (3000) | - |
| 12 | RK-315-A | 100 | 20.684 (3000) | + |
| 13 | RK-315-A | 92 | 20.684 (3000) | + |
| 14 | RK-315-A | 85 | 20.684 (3000) | + |
| 15 | RK-315-A | 80 | 20.684 (3000) | + |
| 16 | RK-315-A | 60 | 20.684 (3000) | + |
| 17 | RK-315-A | 40 | 20.684 (3000) | + |
| 18 | RK-315-A | 13 | 20.684 (3000) | + |
| 19 | RK-315-A | 13 | **13.79 (2000)** | + |
| 20 | RK-315-A | 13 | **6.895 (1000)** | - |